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DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Industrial Application] This invention relates to the manufacturing method of polyvinyl alcohol (it is hereafter written as PVA) system textiles with which protein system natural particles were blended good.

[0002]

[Description of the Prior Art] Conventionally, although PVA system textiles are broadly used as the object for garments, and textiles for industrial materials, on the other hand, various improvement which it is going to bring close to the hygroscopicity of natural fibers, such as wool, silk, cotton, aesthetic property, etc. is performed. Blending natural particles to synthetic fibers including PVA system textiles as the one improvement means is indicated by JP,3-213506,A and JP,3-213507,A. However, for nature system of keratin protein natural particles including wool yarn particles being massive, and existing in a polymer undiluted solution, also in protein system natural particles, etc., even if the dispersibility within PVA system textiles was bad and fibrosed [which is easy to carry out the thread breakage at the time of spinning], there were problems, like intensity is weak.

[0003]

[Problem(s) to be Solved by the Invention] An object of this invention is to have the hygroscopicity which raised the dispersibility in textiles of protein system natural particles, and the above-mentioned problem was solved, and was excellent to the conventional PVA system textiles, and to provide the method of manufacturing the PVA system textiles which also have new aesthetic property and touch.

[0004]

[Means for Solving the Problem] In order to solve the above-mentioned technical problem, this invention has one composition of the following. Namely, a manufacturing method of PVA system textiles carrying out after [addition mixing] spinning of protein system natural particles and the disulfide bond cleavage agent to a PVA system polymer undiluted solution, Or it is a manufacturing method of PVA system textiles carrying out after [addition mixing] spinning of protein system natural particles and the surface-active agent to a PVA system polymer undiluted solution.

[0005] A PVA system polymer used by this invention in partial saponification PVA and a main chain Ethylene, A little olefin system monomers, such as propylene, styrene, acrylic acid and its alkyl ester, methacrylic acid and its alkyl ester, and itaconic acid, can mention a PVA system polymer by which copolymerization was carried out. It is [anything] good if this polymer is dissolved as a solvent of the above-mentioned PVA system polymer, For example, dimethyl sulfoxide, glycerin, ethylene glycol, a diethylene glycol, Polyhydric alcohol classes, such as triethylene glycol, tetraethylene glycol, and trimethylolpropane, Amines, such as ethylenediamine and diethylenetriamine, resorcinol, Solution of mineral salt of lithium halides, such as organic system solvents, such as a formamide and a saturated water solution of urea, or water, a lithium bromide, a lithium chloride, sodium cyanate, zinc chloride, an aluminium chloride, a magnesium chloride, etc. or these mixed solvents can be mentioned.

[0006] With protein system natural particles used by this invention, chemical preparation and/or a

mechanical process grind one or more sorts of natural products containing native proteins, such as albumin, globulin, hemoglobin, casein, collagen, elastin, keratin, and fibroin. Since dispersibility of protein system natural particles [, such as an epithelium of an animal which contains quality of keratin protein especially and hair, a nail, HIZUME, TSUNO, UROKO, and feathers] in textiles improves remarkably by addition of a disulfide bond cleavage agent, they are preferred. Since dispersibility of protein system natural particles with little content of quality of keratin protein in textiles improves by addition of a surface-active agent, they are preferred.

[0007]Particle diameter of these protein system natural particles is 1/10 or less [of a fiber diameter preferably made into the purpose]. When mean particle diameter of this natural particle is made into a micron, it is preferred to pre-use a thing excluding a thing more than 4a micron by filtration etc. Protein system natural particles can be added in the range which does not spoil silk manufacture nature. Hygroscopicity, aesthetic property, etc. are made good and, on the other hand, it is 30 or less % of the weight of 5 % of the weight or more 40 or less % of the weight of 0.5 % of the weight or more from a viewpoint of preventing a textiles physical-properties fall, still more preferably to a PVA system polymer preferably.

[0008]With a disulfide bond cleavage agent used by this invention. Cleave chemically and a disulfide bond in protein specifically Peracetic acid, Reducing agents, such as oxidizers, such as performic acid and hydrogen peroxide, or 2-mercaptoethanol, thioglycolic acid, a sodium sulfide, dithiothreitol, and a dithio ERIS toll, etc. are mentioned, and one sort or two sorts or more may be used together. 2-mercaptoethanol from compatibility, safety, etc. with a solvent of a PVA system polymer and thioglycolic acid are especially preferred.

[0009]An addition in a case of adding a disulfide bond cleavage agent in this invention is 30 or less % of the weight of 0.1 % of the weight or more to an addition of protein system natural particles preferably, and is 20 or less % of the weight of 5 % of the weight or more more preferably. In an addition smaller than 0.1 % of the weight, with an addition whose dispersibility improvement effect in textiles of natural particles is small and which has them, a superfluous disulfide bond cleavage agent remains in textiles, textiles physical properties, such as intensity, may fall, or bleed out may be carried out, after becoming a product. [than 30 % of the weight] [more]

[0010]When adding a disulfide bond cleavage agent in this invention, addition mixing of protein system natural particles and a disulfide bond cleavage agent above-mentioned to the above-mentioned PVA system polymer undiluted solution is carried out. it should just be the stage to carry out addition mixing at the arbitrary times before carrying out spinning -- beforehand -- protein system natural particles -- and, Or addition mixing of the disulfide bond cleavage agent may be carried out at the solvent of a polymer itself, or after mixing and a polymer undiluted solution, and after-mixing spinning of protein system natural particles, a disulfide bond cleavage agent, and the polymer undiluted solution may be carried out simultaneously. [a polymer undiluted solution or a polymer undiluted solution diluted with this solvent, and] Publicly known things, such as a mixing device with impeller, static mixer, and inline blending machine, a roll, the Bambari mixer, a pressurized kneader, a screw type kneading extruder, and a rotor type kneading machine, can be used for a device to mix.

[0011]As a surface-active agent used by this invention, sodium dodecyl sulfate, Sodium dodecyl sulfonate, dodecyl-N-sarcosine sodium, Anionic detergents, such as sodium cholate and a sodium deoxycholate, Cationic surfactants, such as a Sept Iles trimethylammonium star's picture and dodecylpyridinium chloride, 3-[(call amide propyl) dimethylammonio]-1-BUROBAN sulfonic acid, Ampholytic surface active agents, such as BARUMITO yl lysolecithin and dodecyl-beta-alanine, Octyl glycoside, octylthio glycoside, HEBUCHIRU thio glycoside, Decanoly-N-methyl glucamide, polyoxyethylene dodecylether, Although nonionic surfactants, such as polyoxyethylene i-octylphenyl ether, polyoxyethylene nonylphenyl ether, polyoxyethylene fatty acid ester, and polyoxyethylene sorbitol ester, are mentioned, It is preferred to use a nonionic surfactant from a point with forming [little] protein particles, a solvent, etc. and a foreign matter especially.

[0012]An addition in a case of adding the above-mentioned surface-active agent is 10 or less % of the weight of 0.05 % of the weight or more to an addition of protein system natural particles preferably, and

is 5 or less % of the weight of 0.1 % of the weight or more more preferably. in an addition smaller than 0.05 % of the weight, with an addition whose dispersibility improvement effect in textiles of natural particles is small and which has them, a superfluous surface-active agent remains in textiles, and there is a case which came out after textiles physical properties', such as intensity's, having fallen or becoming a product where bleed out is carried out. [than 10 % of the weight] [more] When adding a surface-active agent in this invention, addition mixing of protein system natural particles and a surface-active agent above-mentioned to the above-mentioned PVA system polymer undiluted solution is carried out. it should just be the stage to carry out addition mixing at the arbitrary times before carrying out spinning -- beforehand -- protein system natural particles -- and, Or addition mixing of the surface-active agent may be carried out at the solvent of a polymer itself, or after mixing and a polymer undiluted solution, and after-mixing spinning of protein system natural particles, a surface-active agent, and the polymer undiluted solution may be carried out simultaneously. [a polymer undiluted solution or a polymer undiluted solution diluted with this solvent, and]

[0013] This invention performs spinning of this polymer undiluted solution, after carrying out addition mixing, and it obtains PVA system textiles. A publicly known spinning method is applicable to the technique of fibrosis using the above-mentioned polymer undiluted solution. May carry out the direct inlet (wet spinning) of the regurgitation line of thread into a coagulation bath from a spinneret, and, providing a cap in an about 2-20-mm position on a spinning bath oil level -- the cap -- it is good also by what is called a dryness-and-moisture type spinning method that introduces this spinning undiluted solution breathed out from a hole into a back coagulation bath which made it run minute space between a spinneret hole and a solidified solution side. As a coagulant, it has compatibility to a solvent of a spinning undiluted solution in that case, and a mixed solvent, mineral solution, etc. with a solvent of a thing of a non solvent, for example, methanol, ethanol, acetone, benzene, toluene, or these and a spinning undiluted solution are used to a PVA system polymer. a spinning undiluted solution -- a cap -- it is good also by the spinning [dryly] method for extruding from a hole and evaporating a solvent in a hot wind. It is arbitrary to add colorant, paints, and other publicly known additive agents for PVA system textiles obtained by this invention.

[0014] Although a reason with a disulfide bond cleavage agent effective in improvement in dispersibility of protein system natural particles and protein system particles which especially contain quality of keratin protein is not clear, It acts on a proteinic specific site, and when some protein dissolves into a solvent, condensation between particles is prevented and that whose dispersibility in textiles of these particles improves is presumed. Although a reason with a surface-active agent effective in improvement in dispersibility of protein system particles and protein system particles with little [especially] content of quality of keratin protein is not clear, when a hydrophobic region of a surface-active agent covers a proteinic hydrophobic region, Condensation between particles is prevented and it is presumed whether the dispersibility in textiles of these particles improves.

[0015] Hereafter, an example explains this invention. Raw material composition % in an example means weight %. About particle dispersibility in obtained textiles, when protein system natural particles of a mean-particle-diameter a micron were added, a rate (%) of a particle number which has the particle diameter more than 4a micron among all the particles which exist in textiles was performed by microscope observation of a fiber cross section. If this rate is high, it will judge with particle dispersibility in textiles being bad. Intensity of textiles and moisture absorption asked for equilibrium moisture regain (%) of tensile strength (g/d), temperature of 20**2 **, and 65**2% of relative humidity according to JIS-L-1013, respectively.

[0016]

[Example 1] To the polymer undiluted solution 1000g (20% of polymer concentration) which dissolved in water, PVA (degree-of-polymerization 1800 and degree % of 98 mol of saponification). Addition mixing of what mixed the water 50g, 20g of wool yarn particles (mean particle diameter of 0.5 micron and a thing of 2 microns or more are removed.), and 2 g of 2-mercaptoethanol beforehand is carried out. Spinning and distraction were performed by a publicly known wet spinning method, and PVA system textiles with a single fiber fineness of 5 deniers were obtained. A particle of 2 microns or more was not

observed in obtained textiles. Intensity was 6.5 g/d and moisture absorption was 7.0%. These textiles had soft aesthetic property and a touch which are not in the conventional PVA textiles.

[0017][Comparative example 1] Except not having added wool yarn particles and 2-mercaptoethanol, it carried out like Example 1 and textiles with a single fiber fineness of 5 deniers were obtained. Although intensity was as high as 8.0 g/d, moisture absorption was as low as 4.4%.

[Comparative example 2] Except not having added 2-mercaptoethanol, it carried out like Example 1 and textiles with a single fiber fineness of 5 deniers were obtained. As for the particle dispersibility in the textiles obtained by thread breakage occurring frequently in spinning, 2 microns or more were as bad as 25%, and it was as low as 1.2 g/d. [of intensity]

[0018]

[Example 2] Except having used collagen particles (the mean particle diameter of 0.5 micron and a thing of 2 microns or more are removed.) instead of wool yarn particles, it carried out like Example 1 and textiles with a single fiber fineness of 7 deniers were obtained. A particle of 2 microns or more was not observed in the obtained textiles. Intensity was 6.4 g/d and moisture absorption was 7.2%. These textiles had the soft aesthetic property and touch which are not in the conventional PVA system textiles.

[0019]

[Examples 3-8] Except having changed the addition of wool yarn particles, it carried out like Example 1 and textiles with a single fiber fineness of 5 deniers were obtained. The particle dispersibility of the obtained textiles, intensity, and hygroscopicity were summarized in Table 1.

[0020]

[Example 9] Except having used thioglycolic acid instead of 2-mercaptoethanol, it carried out like Example 1 and textiles with a single fiber fineness of 5 deniers were obtained. A particle of 2 microns or more was not observed in the obtained textiles. Intensity was 7.0 g/d and moisture absorption was 7.0%. These textiles had the soft aesthetic property and touch which are not in the conventional PVA system textiles.

[0021]

[Examples 10-15] Except having changed the addition of 2-mercaptoethanol, it carried out like Example 1 and textiles with a single fiber fineness of 5 deniers were obtained. The particle dispersibility of the obtained textiles, intensity, and hygroscopicity were summarized in Table 2.

[0022]

[Example 16] To the same PVA polymer undiluted solution 1000g as having adjusted in Example 1, beforehand The water 50g, Addition mixing of what mixed 20g of silk particles (the mean particle diameter of 0.5 micron and a thing of 2 microns or more are removed beforehand.) and the polyoxyethylene i-octylphenyl ether 0.2g is carried out. The wet spinning device performed spinning and the distraction, and PVA system textiles with a single fiber fineness of 5 deniers were obtained. A particle of 2 microns or more was not observed in the obtained textiles. Intensity was 6.5 g/d and moisture absorption was 7.0%. These textiles had the soft aesthetic property and touch which are not in the conventional PVA system textiles.

[0023][Comparative example 3] Except not having added silk particles and polyoxyethylene i-octylphenyl ether, it carried out like Example 16 and textiles with a single fiber fineness of 5 deniers were obtained. Although intensity was as high as 8.0 g/d, moisture absorption was as low as 4.4%.

[Comparative example 4] Except not having added polyoxyethylene i-octylphenyl ether, it carried out like Example 16 and textiles with a single fiber fineness of 5 deniers were obtained. As for the particle dispersibility in the textiles obtained by thread breakage occurring frequently in spinning, 2 microns or more were as bad as 25%, and it was as low as 1 and 2 g/d. [of intensity]

[0024]

[Example 17] Except having used collagen particles (the mean particle diameter of 0.5 micron and a thing of 2 microns or more are removed.) instead of silk particles, it carried out like Example 16 and textiles with a single fiber fineness of 7 deniers were obtained. A particle of 2 microns or more was not observed in the obtained textiles. Intensity was 6.5 g/d and moisture absorption was 7.0%. These textiles had the soft aesthetic property and touch which are not in the conventional PVA system textiles.

[0025]

[Examples 18-23] Except having changed the addition of silk particles, it carried out like Example 16 and textiles with a single fiber fineness of 5 deniers were obtained. The particle dispersibility of the obtained textiles, intensity, and hygroscopicity were summarized in Table 3.

[0026]

[Examples 24-27] Except having changed the kind of surface-active agent, it carried out like Example 16 and textiles with a single fiber fineness of 5 deniers were obtained. The particle dispersibility of the obtained textiles, intensity, and hygroscopicity were summarized in Table 4.

[0027]

[Examples 28-33] Except having changed the addition of polyoxyethylene i-octylphenyl ether, it carried out like Example 16 and textiles with a single fiber fineness of 5 deniers were obtained. The particle dispersibility of the obtained textiles, intensity, and hygroscopicity were summarized in Table 5.

[0028]

[Effect of the Invention] In this invention, protein system natural particles distribute good in PVA system textiles so that clearly from an above-mentioned example.

Therefore, there is little thread breakage, the fall of textiles physical properties, such as intensity, is small, and it excels in hygroscopicity, and the PVA system textiles which have the new aesthetic property and touch which are not in the conventional PVA system textiles are obtained.

[0029]

[Table 1]

	ウール微粒子 添加量 (重量%)	繊維中粒子分散性 (粒径4 μ m以上)	強度 (g/d)	吸湿率 (%)
実施例3	0.3	観察されず	7.2	4.8
実施例4	3	観察されず	7.1	5.8
実施例5	7	観察されず	6.7	6.5
実施例6	25	観察されず	6.2	8.0
実施例7	35	観察されず	6.0	8.7
実施例8	45	観察されず	5.8	9.2

[0030]

[Table 2]

	2-メルカプト エタノール 添加量 (重量%)	繊維中粒子分散性 (粒径4 μ ミクロ ン以上)	強 度 (g/d)	吸湿率 (%)
実施例10	0.05	観察されず	6.3	7.0
実施例11	2	観察されず	6.4	7.0
実施例12	7	観察されず	6.5	7.0
実施例13	15	観察されず	6.5	7.0
実施例14	25	観察されず	6.4	7.0
実施例15	35	観察されず	6.2	7.0

[0031]

[Table 3]

	シルク微粒子 添加量 (重量%)	繊維中粒子分散性 (粒径4 μ ミクロ ン以上)	強 度 (g/d)	吸湿率 (%)
実施例18	0.3	観察されず	7.3	4.8
実施例19	3	観察されず	7.2	5.6
実施例20	7	観察されず	6.7	6.5
実施例21	25	観察されず	6.2	8.0
実施例22	35	観察されず	6.0	8.8
実施例23	45	観察されず	5.7	9.2

[0032]

[Table 4]

	界面活性剤 の種類	繊維中粒子分散性 (粒径4 μ ミクロ ン以上)	強 度 (g/d)	吸湿率 (%)
実施例24	(A)	観察されず	6.4	7.0
実施例25	(B)	観察されず	6.4	7.0
実施例26	(C)	観察されず	6.4	7.0
実施例27	(D)	観察されず	6.5	7.0

Note (A):sodium-dodecyl-sulfate (B):Sept Iles trimethylammonium star's-picture (C):BARUMITO yl
lysolecithin (D): Polyoxyethylene sorbitol ether [0033]

[Table 5]

	ポリオキシエチ レンーオクチ ルフェニルエー テル (重量%)	繊維中粒子分散 性 (粒径4 μm クロン以上)	強 度 (g/d)	吸湿率 (%)
実施例 28	0.03	観察されず	6.3	7.0
実施例 29	0.08	観察されず	6.4	7.0
実施例 30	0.3	観察されず	6.5	7.0
実施例 31	3	観察されず	6.5	7.0
実施例 32	8	観察されず	6.4	7.0
実施例 33	15	観察されず	6.3	7.0

[Translation done.]